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RESEARCH MEMORANDUM

EFFECTS OF SOME METAL ADDITIONS ON PROPERTIES OF MOLYBDENUM DISILICIDE

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NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS

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EFFECTS OF SOME METAL ADDITIONS ON PROPERTIES OF MOLYBDENUM DISILICIDE

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SUMMARY

The effect of the addition of approximately 6 percent nickel, cobalt, or platinum on some properties of molybdenum disilicide was investigated. These additions resulted in appreciably lowering the modulus-of-rupture strength from that of unalloyed molybdenum disilicide. The thermal shock resistance was unimproved. The resistance to oxidation was decreased at higher temperatures.

It is believed that above 2000° and 2400° F the nickel and cobalt alloys, respectively, formed low-melting intermetallics and possibly eutectics between these intermetallics. This resulted in considerable porosity. No uncombined metal could be detected in either hot-pressed or heat-treated material. In the platinum-containing bodies, uncombined metal was detected after hot pressing. The concentration of uncombined metal decreased as a result of heat treatment and, after treatment at the higher temperatures, could not be detected.

INTRODUCTION

Because of its excellent resistance to oxidation and its outstanding strength at elevated temperatures, molybdenum disilicide $MoSi_2$ appears to have promise as a material for high-temperature application (ref. 1). Its modulus-of-rupture strength is 90,000 pounds per square inch at 2000° F and the 100-hour life is over 30,000 pounds per square inch at 1800° F (ref. 2). The poor resistance of $MoSi_2$ to thermal shock limits its application.

On the premise that thermal shock resistance can be increased by the introduction of a ductile metal binder, a series of evaluations was conducted to determine the effect of metal additions on the properties of $MoSi_2$.

Nickel, cobalt, and platinum were considered satisfactory for binding and in preliminary tests were found to wet $MoSi_2$.

Preliminary oxidation tests were conducted on hot-pressed bodies of $MoSi_2$ plus 5, 10, and 20 percent of each of these metals. The resistance to oxidation decreased rapidly with increasing metal content. On the basis of these tests, 6 percent was selected as the amount of metal addition for further study.

After these mixtures were hot-pressed, a series of homogenizing treatments was given and their effect on the properties was evaluated. Specimens of each metal addition were given a 20-hour soaking treatment at 2400° F in a helium atmosphere. Density measurements were used to check on the stability of these alloys during this treatment. Lower or higher homogenizing treatments were given to other specimens of each group until the maximum temperature of stability was determined.

MATERIALS

The $MoSi_2$ powder used in this investigation was prepared by the Electro Metallurgical Company.

This powder was ball-milled for 48 hours before it was mixed with the metal powders and for 12 additional hours after it was mixed. All ball milling was done in a steel mill with steel balls, with benzene as a medium. After this treatment the iron content was found to be less than 0.5 percent.

The nickel, cobalt, and platinum powders were obtained from commercial sources.

SPECIMEN PREPARATION

The mixed powders were hot-pressed in graphite dies into 1/2 by 1/2 by $3\frac{1}{4}$ inch bars by the Metal Carbides Corporation at approximately 2800° F under a load of 2500 pounds. The entire cycle required about 45 minutes.

Hot-pressed and homogenized bars were cut in half longitudinally and each half was ground to a finished cross-section of 0.200 by 0.400 inch. This process completely removed the graphite-affected zone. All homogenizing was conducted in a helium atmosphere for 20 hours and the temperatures used were:

Cobalt bearing: 2000° and 2400° F

Nickel bearing: 1800°, 2000°, 2200°, and 2400° F

Platinum bearing: 2400° and 2600° F

Pieces 0.400 inch long were cut from one end of each bar for oxidation and thermal shock evaluations. The long pieces were used for density, resistivity, and modulus-of-rupture evaluations.

POWDER EVALUATION PROCEDURES

Density. - The true densities of the mixed powders were obtained by the pycnometer method with freshly boiled distilled water.

Particle size. - Particle size analysis was conducted with a photelometer as described in reference 3. Xylene was used as the dispersing medium in all analyses reported herein. This procedure was checked by making a partial size analysis of a powder that had previously been analyzed by both the photelometer and sub-sieve techniques.

SPECIMEN EVALUATION PROCEDURES

Density. - The bars were carefully measured and weighed and the density was calculated. Where specimens could not be measured accurately, a thin coating of lacquer was applied and the density was calculated by the water-immersion method.

Resistivity. - The potential drop across a known length was measured; direct current was used. The current was held constant and at a low enough value to avoid heating the piece being tested. A 12-volt storage battery, combined in series with a 5-ohm rheostat, an ammeter (0.1 ampere-divisions), and mercury contacts, was used to provide a constant current. The resistivity was calculated from the measured current, potential drop, and specimen dimensions.

Modulus of rupture. - The modulus-of-rupture evaluations were conducted in a specially adapted Globar furnace as reported in reference 1. The furnace is equipped with a lever-arm system for loading, and constant loading rates are achieved by controlling water flow into a load container.

In all evaluations, a loading rate of 2000 pounds per square inch per minute and a span of 2 inches were used. Each specimen 0.2 by 0.4 inch in cross section was soaked at temperature for 15 minutes prior to loading.

Oxidation resistance. - The specimens approximately 0.400 by 0.400 by 0.200 inch were carefully measured and weighed. Evaluation of oxidation resistance was conducted in a Globar furnace with alundum boats and in still air. All spalled material was included in the weighing. Weight changes per unit of area were recorded and compared.

Thermal shock resistance. - Specimens 0.4 by 0.4 by 0.2 inch were placed in alundum boats and heated to 1800° F in air in a Globar furnace. They were then quenched in water and examined microscopically for cracks. A second cycle from 1800° F, followed by two cycles from 2000°, 2400°, and 2500° F, was repeated until specimen failure.

Metallography. - The microstructure of the hot-pressed alloyed bars was examined, and comparisons to pure $MoSi_2$ structures were made. The effect of soaking time and temperatures was also investigated. The specimens bearing nickel and cobalt were etched electrolytically in a bath of nitric and acetic acids. Those with the platinum addition were etched with aqua regia.

X-ray diffraction. - A wide-range X-ray diffractometer was used to identify the minor phases observed metallographically. Cobalt radiation (iron filtered) was used for the cobalt alloy and copper (nickel filter) radiation for the nickel and platinum alloys.

RESULTS AND DISCUSSION

Density and partical-size analysis. - The powder densities of the mixes by pycnometer measurement were:

Powder	Density, g/ml
$MoSi_2$ + Ni -	6.35
$MoSi_2$ + Co -	6.33
$MoSi_2$ + Pt -	6.98

The particle-size analyses of the mixes showed that the $MoSi_2$ + Co and the $MoSi_2$ + Pt had approximately the same average particle size (2.4 microns), while the average size for the nickel-bearing powder was considerably finer (1.6 microns). These values are the averages of two determinations. The particle-size distribution is given in table I.

Hot-Pressed Bodies

Chemical analysis of bars. - A review of the analyses shown in table II revealed no significant trends when the metal content was correlated with heat-treatment temperature and load. The variation in percentages suggests the possibility of some loss of metal during hot pressing or, more likely, segregation of the powder mixes prior to pressing. However, the differences were believed not so great as to

produce appreciable changes in the physical properties if the bars in themselves were uniform. It was not determined how much the composition varied within a certain bar, but photomicrographs revealed a certain amount of nonuniformity.

Metallographic and X-ray analysis. - The series of photomicrographs in figure 1 represents the structures of $MoSi_2$ plus 6 percent nickel after various heat treatments. In the hot-pressed material (fig. 1(a)) the white matrix is $MoSi_2$ and the grain boundaries contain one or more minor phases. No free nickel could be detected by diffraction techniques, but structures unlike those of nickel or $MoSi_2$ were found to be present. Pfautsch (ref. 4) reported a compound Ni_4Mo_2Si + Ni_3Si_2 and/or Ni_2Si. The grain boundaries in figure 1(d) are multiphase and it is believed that this reaction or a similar one is involved in this alloy. Pfautsch also reported these intermetallics to be low melting, and it is believed that the large increase in porosity or voids as shown by the black areas in figures 1(d) and 1(e) arises from low-melting intermetallics or possibly from low-melting eutectics between the intermetallics present in these alloys.

Figure 2 depicts the structures of hot-pressed and heat-treated $MoSi_2$ plus 6 percent cobalt. Again minor phases were found in the grain boundaries of the $MoSi_2$. These constituents were found to have structures unlike the metal or the $MoSi_2$. No free cobalt could be detected in the hot-pressed or heat-treated bodies by diffraction techniques. The lower porosity suggests that these intermetallics have higher melting temperatures than those found in the nickel-bearing bodies. These phases have not been identified but consistently gave X-ray diffraction lines that increased in intensity as a result of heat treatment, or as heat-treatment temperature was increased.

The structures of the platinum-bearing bodies as hot-pressed and heat-treated are shown in figure 3. As for the other alloys, diffraction techniques showed the presence of intermetallic phases. Free platinum was detected in the hot-pressed bodies but was not found in the heat-treated material. An appreciable densification was measured after the 2600° F heat treatment and is apparent from the photomicrographs.

Density and resistivity. - A review of the densities and resistivities in table III shows that differences existed among the original bars and also between halves of the same bar.

After heat treatment, there were considerable changes in density and resistivity as shown in the graphs in figure 4. The $MoSi_2$ + Pt densities increased slightly with heat-treating temperature while the resistivities decreased considerably.

The densities and resistivities of the cobalt and nickel alloys were affected by heat treatment above 2000° F as shown in table III. Specimens tended to swell and become porous. This, it is believed, arose from low-melting intermetallics and from eutectics between these intermetallics.

Modulus-of-rupture strengths. - When modulus-of-rupture tests were conducted on the $MoSi_2$ + Ni bars, the effect of heat treatment was very apparent as shown in table IV. The 1800° and 2000° F heat treatments were beneficial, whereas heat treatments of 2200° and 2400° F greatly lowered the strengths. At the testing temperature of 2000° F, the best rupture strength was less than 20 percent of the pure $MoSi_2$ strength.

The rupture strengths of the cobalt alloys were much higher than those of the nickel alloys and were not greatly affected by heat treatment up to 2400° F. However, the strengths were appreciably less than those of unalloyed $MoSi_2$ (see table V).

The rupture strengths of the platinum alloys were greatly reduced from those of pure $MoSi_2$ and heat treatment had little effect. At the 2000° F test temperature, the strength was approximately 25 percent of the pure $MoSi_2$ strength (table VI).

In general, hot pressing and heat treatment of $MoSi_2$ plus 6 percent cobalt, nickel, or platinum produces bodies of $MoSi_2$ plus other intermetallics with greatly reduced rupture strengths.

Thermal shock tests. - While the test was rather crude, it served to provide a basis for comparison of the thermal shock resistance of $MoSi_2$ to that of the alloys.

All specimens including pure $MoSi_2$ developed slight surface cracks on the first water quench from 1800° F. Cobalt and nickel specimens cracked appreciably more on subsequent quenches than did the pure $MoSi_2$. Specimens containing platinum developed wider cracks and failed at conditions only slightly less severe than did the pure $MoSi_2$.

The metal additions did not provide sufficient ductile metal binder for the $MoSi_2$. In these hot-pressed bodies, most of the metal had reacted with the $MoSi_2$ to form new intermetallic phases. In view of the condition of the metal additions in the hot-pressed bodies, unimproved thermal shock resistance was not surprising. In general, all alloy bars regardless of heat treatment exhibited less thermal shock resistance than unalloyed $MoSi_2$.

Oxidation resistance. - The air corrosion of pure $MoSi_2$ and of the alloys for 100-hour and 300-hour duration is represented by figures 5(a) and 5(b), respectively.

The $MoSi_2$ + Ni alloys were found to be very unstable above 2000° F. If the heat treatment or the test temperature exceeded 2000° F, the bodies became porous and oxidized badly.

The air corrosion rates of the cobalt and platinum alloys are higher than that of pure $MoSi_2$ at 2450° F. Distortion of the cobalt- and platinum-bearing bodies occurred above 2450° F.

SUMMARY OF RESULTS

The effect of the addition of approximately 6 percent of nickel, cobalt, or platinum on the properties of molybdenum disilicide was investigated and results were as follows:

1. The modulus-of-rupture strength of hot-pressed bodies of molybdenum disilicide plus 6 percent nickel was about one-third that of pure molybdenum disilicide. Intermetallics that are believed to be low melting were found to be present in this material and no uncombined nickel could be detected. This material was found to be stable up to 2000° F. Above this temperature, these low-melting intermetallics and possibly eutectics between these intermetallics produced considerable porosity. As a result, the oxidation resistance and strength were very low. The thermal shock resistance was unimproved over that of unalloyed $MoSi_2$.

2. The modulus-of-rupture strength of hot-pressed cobalt-bearing material was found to be considerably better than that of the nickel alloy. No uncombined metal could be detected in either hot-pressed or heat-treated material. The oxidation resistance was also found to be only slightly less than that of pure $MoSi_2$ up to 2200° F.

A sharp decrease in density and oxidation resistance resulted whenever this material was heated above 2400° F. It is believed that the intermetallics and eutectics in this material melt at higher temperatures than those found in the nickel alloy. No appreciable difference in the thermal-shock resistance over that of unalloyed molybdenum disilicide was found.

3. Hot-pressed bodies of molybdenum disilicide plus 6 percent platinum were found to have rupture strengths very much less than pure molybdenum disilicide. The oxidation resistance of this material was found to compare with pure molybdenum disilicide up to 2000° F but decreased considerably at higher temperatures. Uncombined platinum was detected in the hot-pressed material, but decreased and finally was all combined in new structures at the higher heat-treatment temperatures. No difference in thermal shock resistance was found.

4. In general, in all three systems involved, the metal added cannot exist in equilibrium with molybdenum disilicide. A more careful screening involving more than wetting is necessary to find metals that will provide a ductile binding for molybdenum disilicide.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, February 23, 1954

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2. Long, Roger A.: Fabrication and Properties of Hot-Pressed Molybdenum Disilicide. NACA RM E50F22, 1950.

3. States, M. N.: Specific Surface and Particle Size Distribution of Finely Divided Materials. A.S.T.M. Proc., vol 39, 1939, p. 795.

4. Pfautsch: Über das Dreistoffsystem Mo-Ni-Si. Zeit. für Metallkunde. Bd. 17, 1925, pp. 48-52.

TABLE I. - PARTICLE SIZE DISTRIBUTION, PERCENT BY WEIGHT

Fraction, microns	$MoSi_2$ + Ni		$MoSi_2$ + Co		$MoSi_2$ + Pt	
	Test 1	Test 2	Test 1	Test 2	Test 1	Test 2
0-1	21.6	25.8	9.7	11.6	8.2	5.6
1-2	40.4	38.0	25.1	27.8	31.1	25.5
2-3	14.8	16.4	34.4	28.1	30.3	31.1
3-4	7.3	8.0	16.2	21.4	14.7	21.0
4-5	9.8	5.3	10.3	11.1	6.6	8.7
5-6	6.1	6.5	4.3		4.1	3.7
6-7					5.0	4.4

TABLE II. - ADDITIONAL METAL CONTENT OF TEST BARS BY CHEMICAL ANALYSIS

Sample number	Nickel, percent	Sample number	Cobalt, percent	Sample number	Platinum, percent
8A	5.56	6A	6.48	1B	5.31
A2	5.79	1	6.26	10	5.06
B2	5.89	4	5.94	5	4.03
D3	4.69			11	5.77
C1	6.00				

TABLE III. - DENSITY AND RESISTIVITY OF MOLYBDENUM DISILICIDE PLUS 6 PERCENT METALLIC ADDITIVE

(a) Nickel.

Sample number	Heat-treating temperature, °F	Density, g/ml	Resistivity, microhm-cm
1A	None	5.90	53.71
1B	None	5.91	57.94
2A	None	5.99	37.49
3A	None	5.84	65.98
3B	None	5.90	57.20
av.		5.91	54.46
1	2400	6.05	28.7
2	2400	5.76	50.7
3	2400	5.88	44.0
4	2400	5.84	44.3
5	2400	5.88	45.6
6	2400	6.02	28.7
7	2400	6.02	29.1
8	2400	5.91	45.5
9	2400	5.81	47.9
10	2400	5.99	30.2
11	2600	6.13	28.7
12	2600	5.96	46.7
13	2600	6.15	28.4
14	2600	5.88	45.0
av.	2400	5.92	39.5
av.	2600	6.03	37.2

TABLE III. - Continued. DENSITY AND RESISTIVITY OF MOLYBDENUM DISILICIDE PLUS 6 PERCENT METALLIC ADDITIVE

(b) Cobalt.

Sample number	Heat-treating temperature, °F	Density, g/ml	Resistivity, microhm-cm
4A	None	5.97	44.91
4B	None	5.98	42.93
5A	None	5.99	42.17
5B	None	5.97	39.69
6A	None	6.01	43.62
6B	None	5.98	40.72
av.		5.98	42.34
1	2000	5.99	39.1
2	2000	5.97	37.0
3	2000	5.91	38.0
4	2000	5.92	41.3
av.		5.95	38.8
1	2400	5.82	40.92
2	2400	5.90	37.12
3	2400	5.99	39.33
4	2400	5.83	40.55
5	2400	5.67	43.52
6	2400	5.69	47.73
7	2400	5.87	40.28
8	2400	5.71	46.35
9	2400	5.89	38.04
10	2400	5.97	36.01
11	2400	5.84	41.41
12	2400	5.82	40.90
av.		5.83	41.01

TABLE III. - Concluded. DENSITY AND RESISTIVITY OF MOLYBDENUM DISILICIDE PLUS 6 PERCENT METALLIC ADDITIVE

(c) Platinum.

Sample number	Heat-treating temperature, °F	Density, g/ml	Resistivity, microhm-cm
7A	None	5.84	46.05
7B	None	5.91	42.32
8A	None	5.91	42.26
8B	None	5.80	48.11
9A	None	5.96	39.61
9B	None	5.98	38.06
av.		5.90	42.73
A1	1800	5.92	41.3
A2	1800	5.94	42.5
A3	1800	5.97	41.0
A4	1800	5.91	41.8
av.		5.94	41.7
B1	2000	5.83	42.7
B2	2000	5.89	40.0
B3	2000	5.86	41.9
B4	2000	5.75	44.8
av.		5.83	42.3
D1	2200	5.32	45.4
D2	2200	5.62	39.3
D3	2200	5.22	44.7
D4	2200	5.06	51.3
av.		5.32	45.2
C1	2400	5.20	44.5
C2	2400	5.03	45.4
C3	2400	4.71	50.7
C4	2400	4.40	61.1
av.		4.83	50.4

TABLE IV. - MODULUS-OF-RUPTURE STRENGTHS OF MOLYBDENUM DISILICIDE PLUS 6 PERCENT NICKEL

Test temperature, °F	Sample number	Resistivity, microhm-cm	Density, g/ml	Modulus of rupture, psi	Heat treatment		
					Temperature, °F	Time, hr	Atmosphere
Room	8A	42.26	5.95	38,600	None		
Room	A2	42.50	5.94	49,700	1800	-20	Helium
Room	B2	40.00	5.89	37,200	2000	-20	Helium
Room	D3	44.70	5.22	24,600	2200	-20	Helium
Room	C1	44.50	5.20	25,000	2400	-20	Helium
1800	7B	42.32	5.91	25,500	None		
1800	A1	41.30	5.92	34,500	1800	-20	Helium
1800	B1	42.70	5.83	35,000	2000	-20	Helium
1800	D1	45.40	5.32	27,500	2200	-20	Helium
1800	C2	45.40	5.03	17,000	2400	-20	Helium
2000	9A	39.61	5.88	3,650	None		
2000	A3	41.00	5.97	13,700	1800	-20	Helium
2000	B3	41.90	5.86	14,800	2000	-20	Helium
2000	D2	39.30	5.62	6,560	2200	-20	Helium
2000	C3	50.70	4.71	3,000	2400	-20	Helium
2000	[a]2	20.50	6.07	86,700			

[a]Pure molybdenum disilicide.

TABLE V. - MODULUS-OF-RUPTURE STRENGTHS OF MOLYBDENUM DISILICIDE PLUS 6 PERCENT COBALT

Test temperature, °F	Sample number	Resistivity, microhm-cm	Density, g/ml	Modulus of rupture, psi	Heat treatment		
					Temperature, °F	Time, hr	Atmosphere
Room	6A	43.62	5.94	60,000	None		
Room	1	39.10	5.99	48,900	2000	-20	Helium
Room	4	40.55	5.83	43,500	2400	-20	Helium
1800	5A	42.17	6.01	75,000	None		
1800	2	37.00	5.97	76,100	2000	-20	Helium
1800	1	40.92	5.82	64,200	2400	-20	Helium
2000	5B	39.69	5.99	62,100	None		
2000	3	38.00	5.91	60,300	2000	-20	Helium
2000	12	40.90	5.82	53,800	2400	-20	Helium
2000	[a]2	20.50	6.07	86,700			

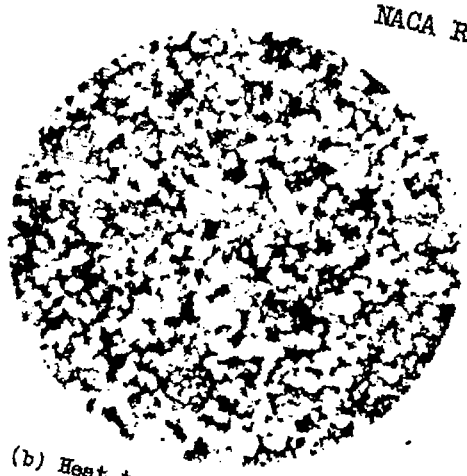
[a]Pure molybdenum disilicide.

TABLE VI. - MODULUS-OF-RUPTURE STRENGTHS OF MOLYBDENUM DISILICIDE PLUS 6 PERCENT PLATINUM

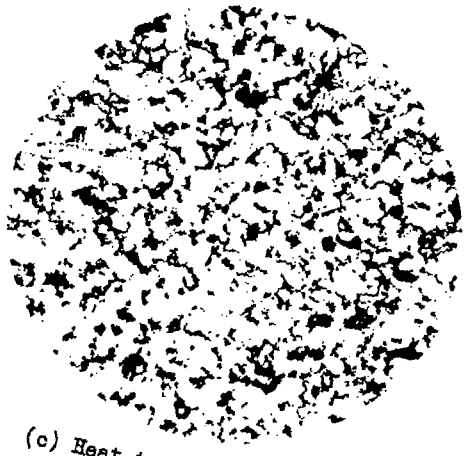
Test temperature, °F	Sample number	Resistivity, microhm-cm	Density, g/ml	Modulus of rupture, psi	Heat treatment		
					Temperature, °F	Time, hr	Atmosphere
Room	1B	57.94	5.92	41,700	None		
Room	10	30.20	5.99	53,600	2400	-20	Helium
Room	5	45.60	5.88	49,800	2400	-20	Helium
Room	11	28.70	6.13	43,700	2600	-20	Helium
1800	1A	53.71	5.89	39,500	None		
1800	1	28.70	6.05	40,900	2400	-20	Helium
1800	3	44.00	5.88	40,100	2400	-20	Helium
1800	12	46.70	5.96	35,900	2600	-20	Helium
2000	3B	57.20	5.91	14,900	None		
2000	6	28.70	6.02	24,700	2400	-20	Helium
2000	4	44.30	5.84	18,750	2400	-20	Helium
2000	13	28.40	6.15	27,600	2600	-20	Helium
2000	[a]2	20.50	6.07	86,700			

[a]Pure molybdenum disilicide.

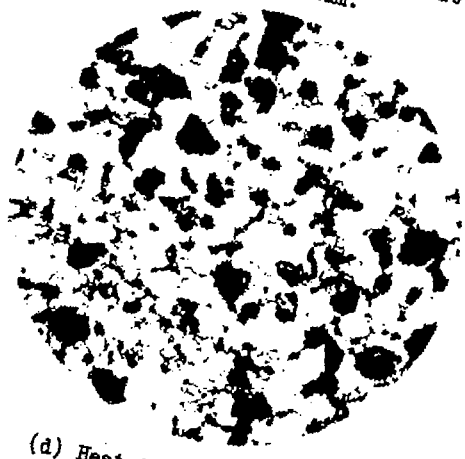
(a) Hot pressed.



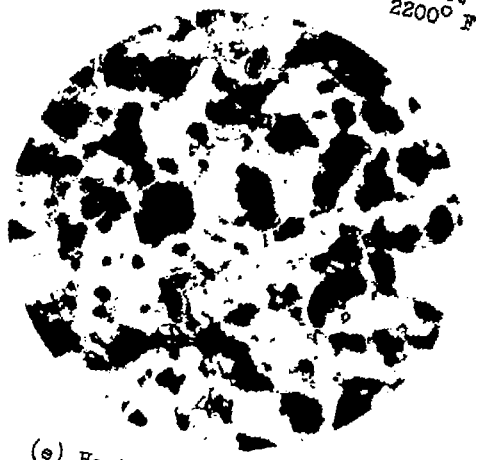
(b) Heat treated 20 hours at 1800° F in helium.



(c) Heat treated 20 hours at 2000° F in helium.



(d) Heat treated 20 hours at 2200° F in helium.



(e) Heat treated 20 hours at 2400° F in helium.

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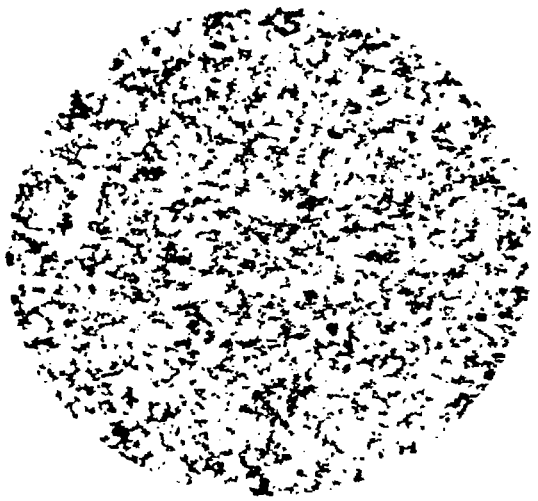
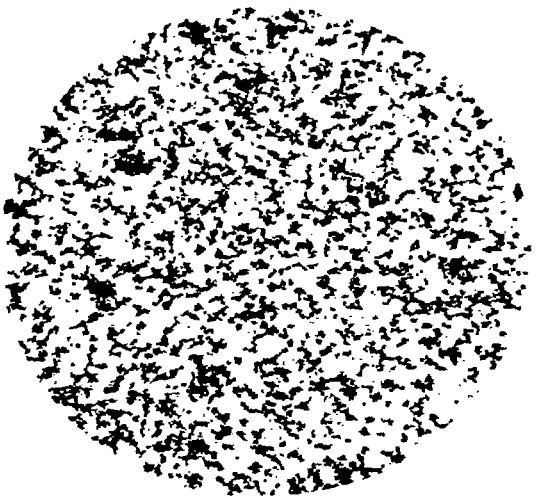
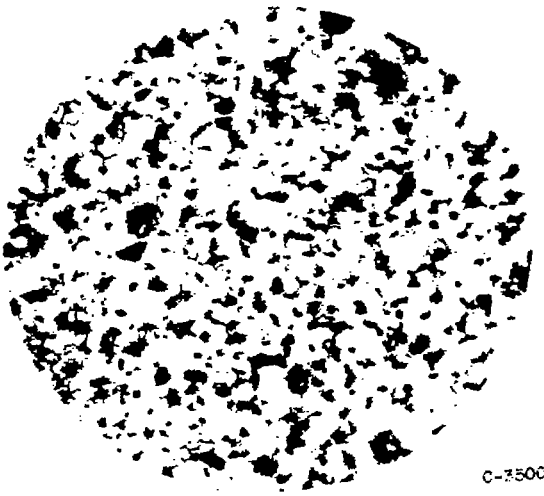
Figure 1. - Structure of molybdenum disilicide plus 6 percent nickel. X500. Electrolytically etched with nitric and acetic acids.



(a) Hot pressed.



(b) Heat treated 20 hours at 2000° F in helium.



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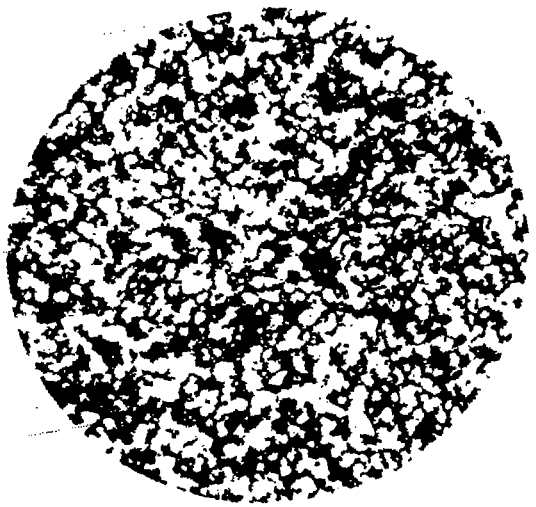
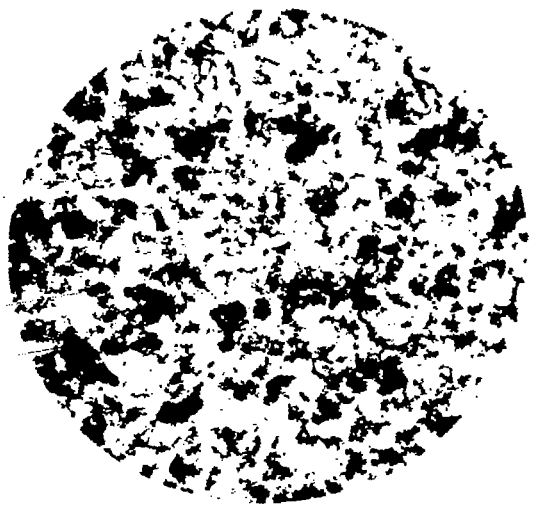
(c) Heat treated 20 hours at 2400° F in helium.

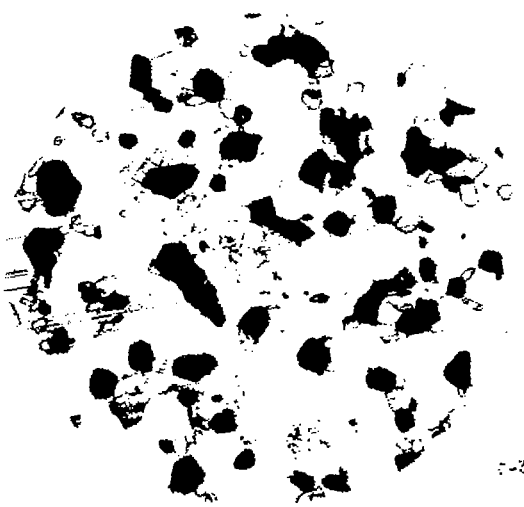
Figure 2. - Structure of molybdenum disilicide plus 6 percent cobalt. X1000. Electrolytically etched with nitric and acetic acids.



(a) Hot pressed.

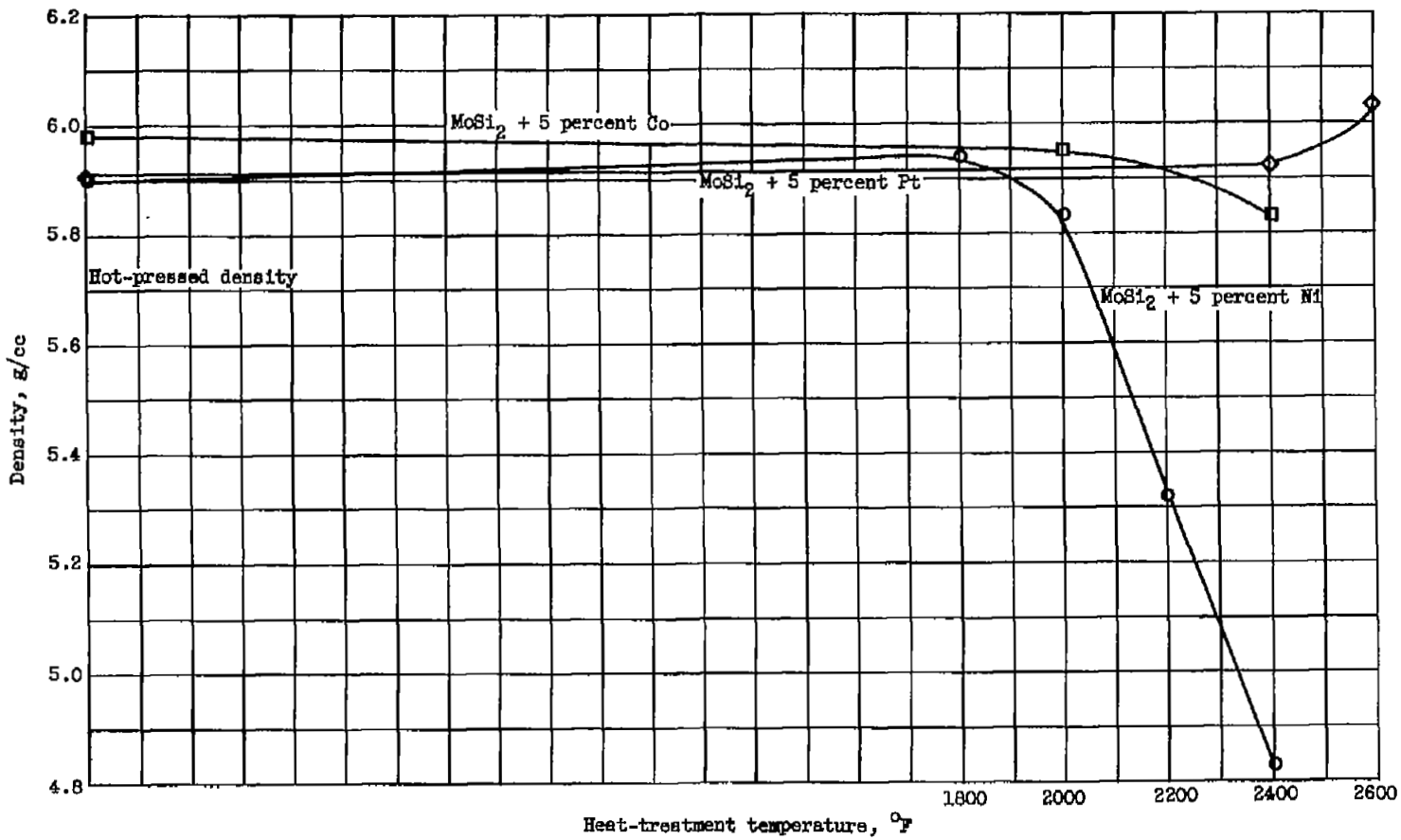


(b) Heat treated 20 hours at 2400° F in helium.



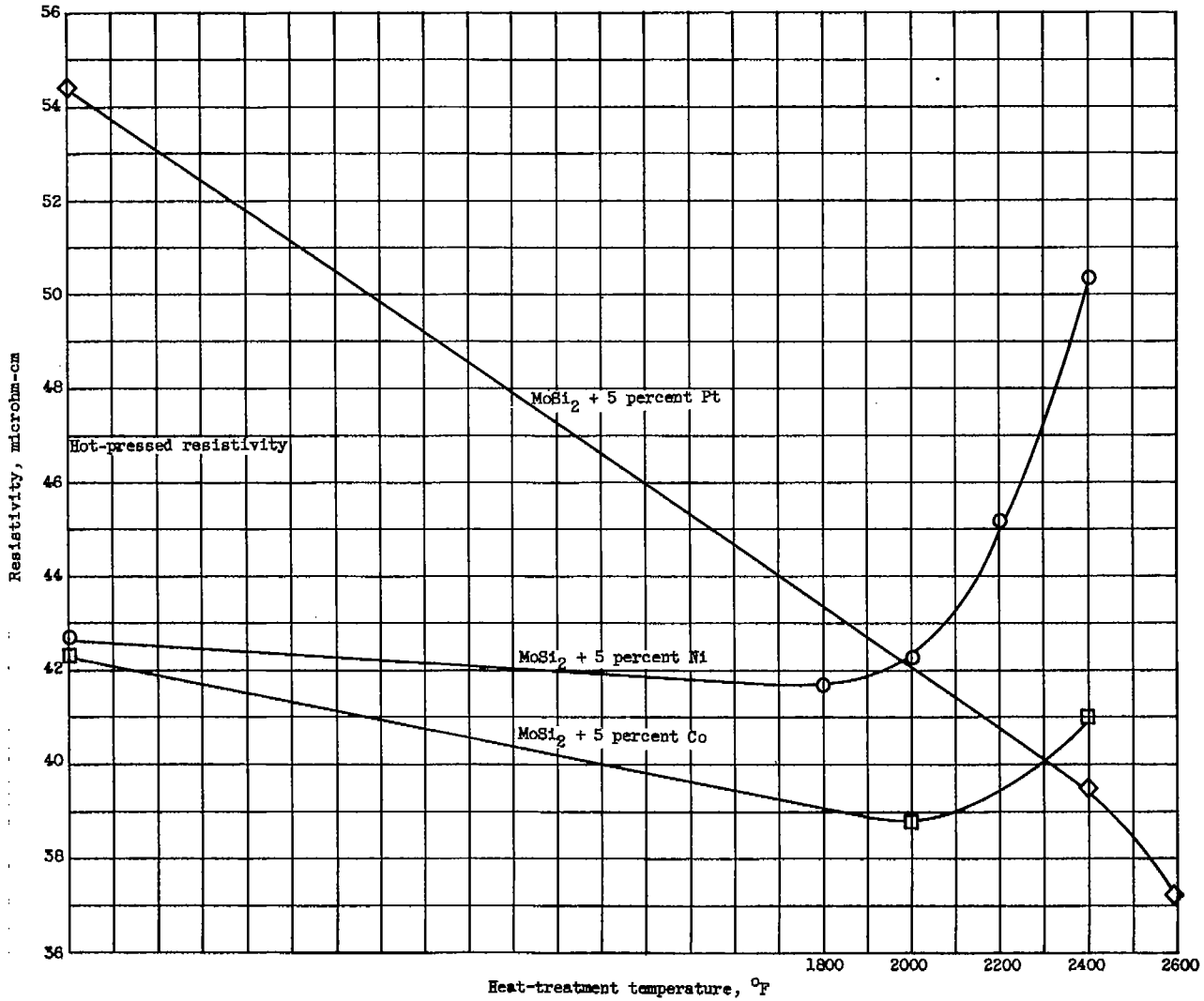
(c) Heat treated 20 hours at 2600° F in helium.

Figure 3. - Structure of molybdenum disilicide plus 6 percent platinum. X500. Aqua regia etch.



(a) Density.

Figure 4. - Effect of heat treatment on properties of molybdenum disilicide alloys.



(b) Resistivity.

Figure 4. - Effect of heat treatment on properties of molybdenum disilicide alloys.

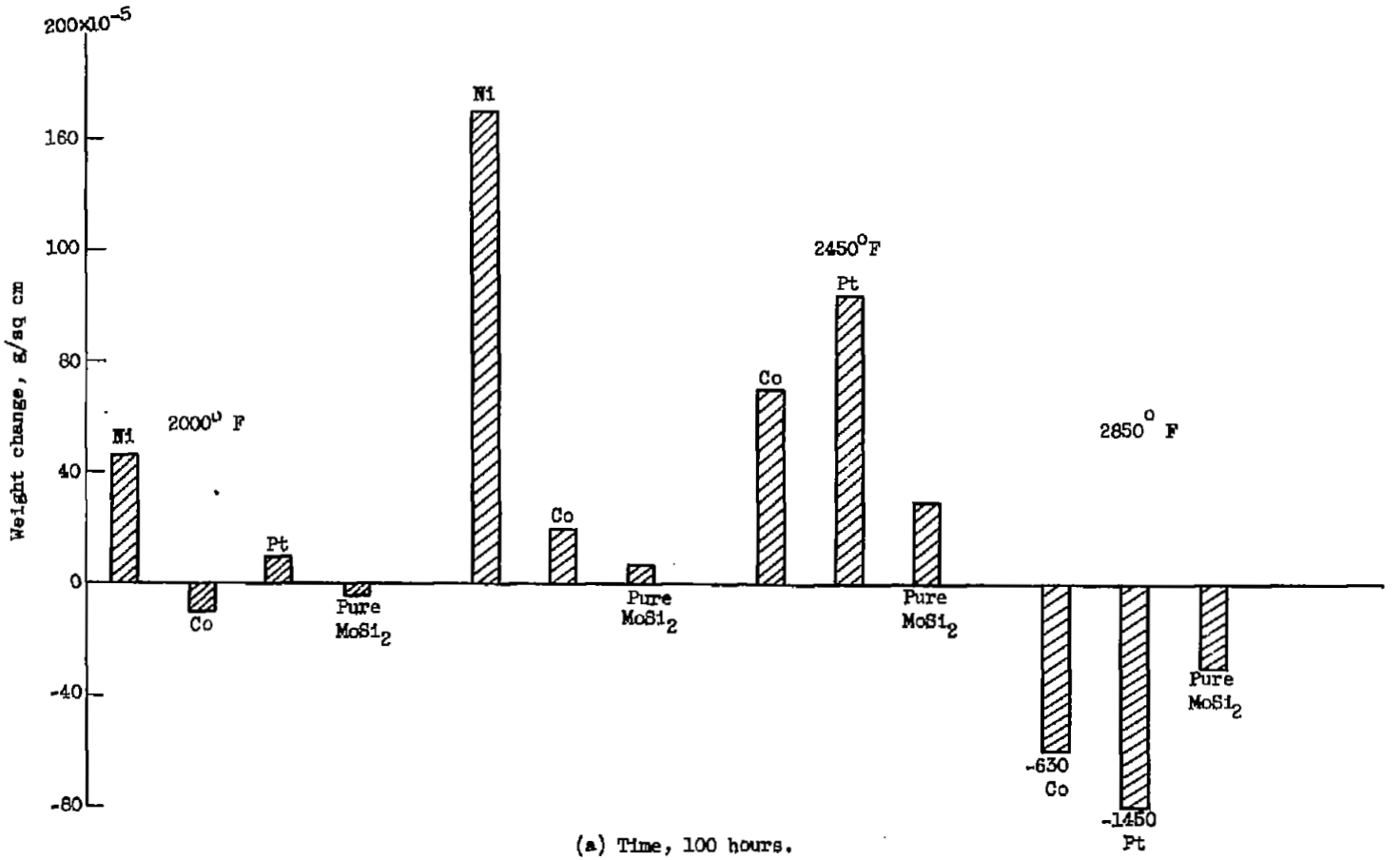
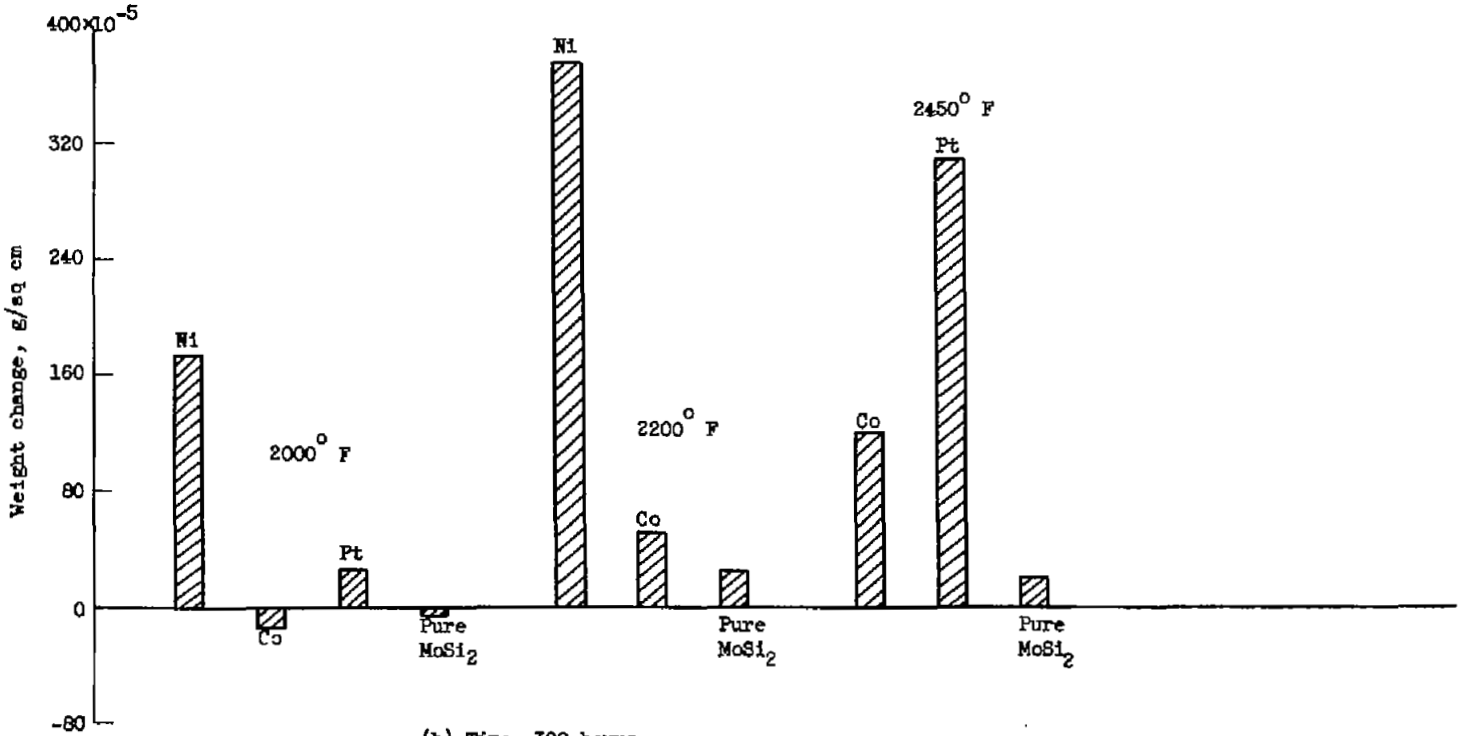


Figure 5. - Air corrosion of pure molybdenum disilicide and alloys. (Values for pure molybdenum disilicide: at 2000° F, from ref. 1; at 2200° and 2450° F, from ref. 2.)



(b) Time, 300 hours.

Figure 5. - Air corrosion of pure molybdenum disilicide and alloys. (Values for pure molybdenum disilicide: at 2000° F, from ref. 1; at 2200° and 2450° F, from ref. 2.)